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Crystal structure of 5,11-dihydropyrido-[2,3-*b*][1,4]benzodiazepin-6-one

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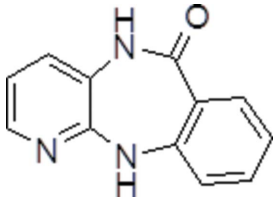
The title compound, $C_{12}H_9N_3O$, is an intermediate in the synthesis of the muscarinic M2 receptor antagonist AFDX-384. The seven-membered ring adopts a boat conformation and the dihedral angle between the planes of the aromatic rings is 41.51 (9)°. In the crystal, molecules are linked into [001] chains of alternating inversion dimers formed by pairs of N—H···O hydrogen bonds and pairs of N—H···N hydrogen bonds. In both cases, $R_2^2(8)$ loops are generated.

Keywords: crystal structure; pyridobenzodiazepine; boat conformation; hydrogen bonding.

CCDC reference: 1024195

1. Related literature

For the synthesis of the title compound, see: Holzgrabe & Heller (2003). For the biological activity of substituted 5,11-dihydropyrido[2,3-*b*][1,4]benzodiazepin-6-ones, see: Mohr *et al.* (2004); Tahtaoui *et al.* (2004).



2. Experimental

2.1. Crystal data

$C_{12}H_9N_3O$
M_r = 211.22
Triclinic, $P\overline{1}$
a = 3.7598 (5) Å
b = 10.2467 (14) Å
c = 12.8768 (17) Å
α = 104.628 (6)°
β = 96.616 (5)°
γ = 98.009 (4)°
V = 469.43 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.35 × 0.26 × 0.06 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.898, T_{max} = 0.959
6425 measured reflections
2000 independent reflections
1467 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
S = 1.06
2000 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.87 (2)	1.98 (2)	2.840 (2)	175 (2)
N3—H3···N1^{ii}	0.93 (2)	2.28 (2)	3.200 (2)	168.7 (19)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+2$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *OLEX2*.solve (Bourhis *et al.*, 2015); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*, *Mercury* (Macrae *et al.*, 2006) and *enCIFer* (Allen *et al.*, 2004).

Acknowledgements

The authors thank Andreas Lorbach and Todd B. Marder (Institute of Inorganic Chemistry, Wuerzburg University) for the data collection and structure solution. We appreciate the financial support provided to NMR by the Deutscher Akademischer Austauschdienst (DAAD). Thanks are also due to the Deutsche Forschungsgemeinschaft for financial support (SFB 630, Recognition, Preparation and Functional Analysis of Agents against Infectious Diseases, project A1).

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7396).

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supporting information

Acta Cryst. (2015). E**71**, o304–o305 [https://doi.org/10.1107/S2056989015006817]

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S1. Experimental

The title compound was synthesized as previously reported (Holzgrabe & Heller, 2003) and recrystallized from methanol–toluene.

S2. Refinement

The N- and C-bound H atoms were included in calculated positions and refined as riding: N2—H = 0.86 Å, C—H and N3—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(C).

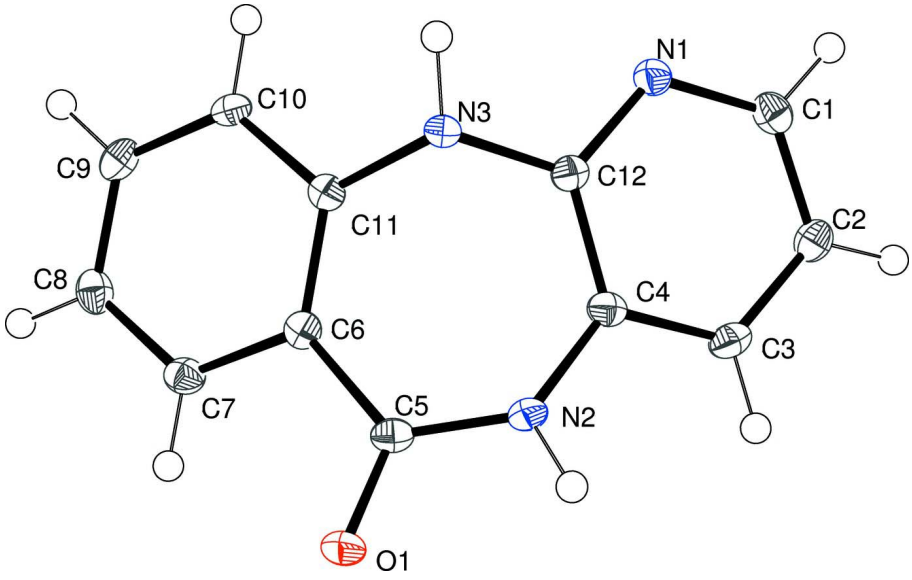

Figure 1

ORTEP drawing of the title compound showing atom labeling and 50% probability displacement ellipsoids.

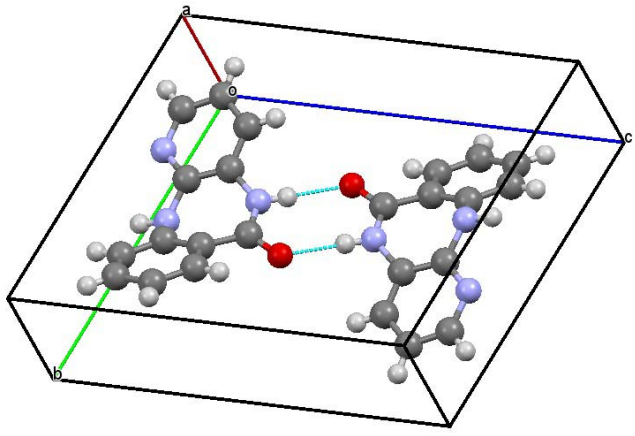

Figure 2

Unit-cell packing of the title compound showing two inverted molecules linked by hydrogen bonds indicated as dotted lines.

5,11-Dihydropyrido[2,3-*b*][1,4]benzodiazepin-6-one

Crystal data

$C_{12}H_9N_3O$
$M_r = 211.22$
Triclinic, $P\overline{1}$
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$c = 12.8768$ (17) Å
$\alpha = 104.628$ (6)°
$\beta = 96.616$ (5)°
$\gamma = 98.009$ (4)°
$V = 469.43$ (11) Å^3
$Z = 2$
$F(000) = 220$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1512 reflections
$\theta = 2.3$–$26.2°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Plate, colourless
0.35 × 0.26 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{min} = 0.898$, $T_{max} = 0.959$
6425 measured reflections
2000 independent reflections
1467 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 26.8°$, $\theta_{min} = 1.7°$
$h = -4 \rightarrow 4$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.110$
$S = 1.06$
2000 reflections

153 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.2092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. Absorption correctiuon: SADABS-2012/1 (Bruker,2012) was used for absorption correction. wR2(int) was 0.0475 before and 0.0419 after correction. The Ratio of minimum to maximum transmission is 0.9367. The $\lambda/2$ correction factor is 0.0015.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3371 (4)	0.33046 (14)	0.49729 (10)	0.0196 (3)
N3	0.4460 (4)	0.42582 (15)	0.83790 (13)	0.0152 (4)
N1	0.3852 (4)	0.64248 (15)	0.93418 (12)	0.0157 (4)
N2	0.3945 (4)	0.51349 (17)	0.63963 (13)	0.0163 (4)
C4	0.3379 (5)	0.59565 (19)	0.74023 (14)	0.0143 (4)
C12	0.3836 (5)	0.55643 (19)	0.83732 (14)	0.0133 (4)
C1	0.3340 (5)	0.7706 (2)	0.93750 (16)	0.0174 (4)
H1	0.3431	0.8320	1.0051	0.021*
C10	0.0818 (5)	0.20571 (19)	0.81375 (15)	0.0148 (4)
H10	0.1291	0.2203	0.8887	0.018*
C3	0.2728 (5)	0.72576 (19)	0.74596 (15)	0.0168 (4)
H3A	0.2314	0.7535	0.6829	0.020*
C6	0.1477 (5)	0.28402 (18)	0.65494 (14)	0.0137 (4)
C7	−0.0602 (5)	0.15952 (19)	0.59213 (15)	0.0162 (4)
H7	−0.1071	0.1434	0.5171	0.019*
C11	0.2231 (5)	0.30746 (18)	0.76774 (14)	0.0130 (4)
C5	0.2998 (5)	0.37759 (19)	0.59331 (14)	0.0150 (4)
C2	0.2691 (5)	0.81567 (19)	0.84655 (15)	0.0170 (4)
H2A	0.2239	0.9039	0.8521	0.020*
C8	−0.1979 (5)	0.0597 (2)	0.63858 (16)	0.0177 (4)
H8	−0.3366	−0.0226	0.5956	0.021*
C9	−0.1254 (5)	0.08462 (19)	0.75055 (15)	0.0168 (4)
H9	−0.2180	0.0187	0.7831	0.020*
H3	0.497 (6)	0.419 (2)	0.9087 (19)	0.027 (6)*
H2	0.467 (6)	0.557 (2)	0.5943 (19)	0.031 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0270 (8)	0.0201 (7)	0.0118 (7)	0.0026 (6)	0.0058 (6)	0.0042 (6)
N3	0.0192 (9)	0.0143 (8)	0.0113 (8)	0.0023 (7)	−0.0008 (7)	0.0036 (7)

N1	0.0174 (8)	0.0157 (8)	0.0137 (8)	0.0016 (7)	0.0032 (6)	0.0039 (7)
N2	0.0212 (9)	0.0165 (9)	0.0122 (8)	0.0010 (7)	0.0043 (7)	0.0062 (7)
C4	0.0119 (9)	0.0174 (10)	0.0125 (9)	−0.0007 (8)	0.0012 (7)	0.0041 (8)
C12	0.0101 (9)	0.0155 (10)	0.0143 (9)	−0.0002 (7)	0.0015 (7)	0.0053 (8)
C1	0.0159 (10)	0.0178 (10)	0.0173 (10)	0.0024 (8)	0.0046 (8)	0.0018 (8)
C10	0.0158 (9)	0.0184 (10)	0.0128 (9)	0.0052 (8)	0.0031 (7)	0.0072 (8)
C3	0.0153 (10)	0.0203 (10)	0.0166 (10)	0.0021 (8)	0.0011 (8)	0.0097 (8)
C6	0.0117 (9)	0.0151 (10)	0.0154 (9)	0.0038 (8)	0.0046 (7)	0.0043 (8)
C7	0.0150 (10)	0.0192 (10)	0.0145 (9)	0.0047 (8)	0.0005 (7)	0.0044 (8)
C11	0.0105 (9)	0.0148 (9)	0.0139 (9)	0.0040 (7)	0.0027 (7)	0.0032 (8)
C5	0.0135 (9)	0.0193 (10)	0.0127 (9)	0.0029 (8)	0.0013 (7)	0.0056 (8)
C2	0.0160 (10)	0.0163 (10)	0.0209 (10)	0.0047 (8)	0.0045 (8)	0.0071 (9)
C8	0.0139 (10)	0.0151 (10)	0.0215 (10)	0.0012 (8)	0.0004 (8)	0.0024 (8)
C9	0.0135 (9)	0.0175 (10)	0.0229 (11)	0.0039 (8)	0.0054 (8)	0.0102 (9)

Geometric parameters (Å, º)

O1—C5	1.240 (2)	C10—C11	1.396 (3)
N3—C12	1.392 (2)	C10—C9	1.372 (3)
N3—C11	1.406 (2)	C3—H3A	0.9300
N3—H3	0.93 (2)	C3—C2	1.390 (3)
N1—C12	1.332 (2)	C6—C7	1.396 (3)
N1—C1	1.344 (2)	C6—C11	1.399 (2)
N2—C4	1.412 (2)	C6—C5	1.487 (3)
N2—C5	1.347 (2)	C7—H7	0.9300
N2—H2	0.87 (3)	C7—C8	1.380 (3)
C4—C12	1.406 (3)	C2—H2A	0.9300
C4—C3	1.374 (3)	C8—H8	0.9300
C1—H1	0.9300	C8—C9	1.387 (3)
C1—C2	1.372 (3)	C9—H9	0.9300
C10—H10	0.9300		

C12—N3—C11	121.58 (15)	C7—C6—C11	119.17 (17)
C12—N3—H3	110.9 (13)	C7—C6—C5	115.70 (16)
C11—N3—H3	112.7 (13)	C11—C6—C5	124.91 (17)
C12—N1—C1	117.87 (16)	C6—C7—H7	119.2
C4—N2—H2	115.9 (15)	C8—C7—C6	121.68 (18)
C5—N2—C4	130.98 (17)	C8—C7—H7	119.2
C5—N2—H2	112.2 (15)	C10—C11—N3	117.55 (16)
C12—C4—N2	123.05 (17)	C10—C11—C6	118.58 (17)
C3—C4—N2	118.46 (17)	C6—C11—N3	123.83 (17)
C3—C4—C12	118.12 (17)	O1—C5—N2	119.17 (17)
N3—C12—C4	121.47 (16)	O1—C5—C6	119.73 (17)
N1—C12—N3	115.93 (16)	N2—C5—C6	121.09 (16)
N1—C12—C4	122.55 (17)	C1—C2—C3	118.16 (18)
N1—C1—H1	118.2	C1—C2—H2A	120.9
N1—C1—C2	123.52 (18)	C3—C2—H2A	120.9
C2—C1—H1	118.2	C7—C8—H8	120.7

C11—C10—H10	119.4	C7—C8—C9	118.69 (18)
C9—C10—H10	119.4	C9—C8—H8	120.7
C9—C10—C11	121.27 (17)	C10—C9—C8	120.60 (18)
C4—C3—H3A	120.2	C10—C9—H9	119.7
C4—C3—C2	119.64 (17)	C8—C9—H9	119.7
C2—C3—H3A	120.2		

N1—C1—C2—C3	−3.0 (3)	C7—C6—C11—C10	−1.0 (3)
N2—C4—C12—N3	−7.9 (3)	C7—C6—C5—O1	−22.0 (3)
N2—C4—C12—N1	169.29 (18)	C7—C6—C5—N2	157.23 (17)
N2—C4—C3—C2	−170.60 (17)	C7—C8—C9—C10	−0.5 (3)
C4—N2—C5—O1	170.53 (18)	C11—N3—C12—N1	132.19 (18)
C4—N2—C5—C6	−8.7 (3)	C11—N3—C12—C4	−50.5 (2)
C4—C3—C2—C1	0.4 (3)	C11—C10—C9—C8	0.5 (3)
C12—N3—C11—C10	−129.09 (19)	C11—C6—C7—C8	1.0 (3)
C12—N3—C11—C6	53.5 (2)	C11—C6—C5—O1	152.47 (18)
C12—N1—C1—C2	2.1 (3)	C11—C6—C5—N2	−28.3 (3)
C12—C4—C3—C2	2.7 (3)	C5—N2—C4—C12	42.4 (3)
C1—N1—C12—N3	178.56 (16)	C5—N2—C4—C3	−144.7 (2)
C1—N1—C12—C4	1.3 (3)	C5—C6—C7—C8	175.82 (17)
C3—C4—C12—N3	179.21 (17)	C5—C6—C11—N3	2.0 (3)
C3—C4—C12—N1	−3.6 (3)	C5—C6—C11—C10	−175.34 (17)
C6—C7—C8—C9	−0.2 (3)	C9—C10—C11—N3	−177.23 (16)
C7—C6—C11—N3	176.35 (17)	C9—C10—C11—C6	0.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.87 (2)	1.98 (2)	2.840 (2)	175 (2)
N3—H3···N1^{ii}	0.93 (2)	2.28 (2)	3.200 (2)	168.7 (19)

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